

Composites of wood and *trans*-1,4-isoprene rubber II: Processing conditions for production of the composites

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Abstract The occurrence of grafting between WF and MTIR through esterification, the processing conditions for production of the composites, and the effect of MAH concentration on some physical and mechanical properties as well as the biodegradability of the composites have been studied. The FTIR measurement demonstrated that grafting through esterification had been attained between OH-groups of WF and acid anhydride groups of MTIR during kneading. The optimum kneading conditions for preparing the composites were 120 °C, 30 ~ 90 rpm for 10 min. The tensile properties of the composites with WF based filler are greater than those with CS (corn starch) based filler. Addition of 10% of MTIR•10 and 8% MAH prepared using the *two-step* and *one-step* process, respectively to the TIR-WF composites gave the best result of tensile properties. The presence of MAH in the TIR-WF composites also improved the dimensional stabilization of the resulting composites. The tensile strength of the composites prepared using the *one-step* process was about 90% of the composite prepared using the *two-step* process. The composites of TIR-WF with MTIR compatibilizer had slightly less resistance against subterranean termite (*C. formosanus*), brown-rot fungus (*T. palustris*), and white-rot fungus (*C. versicolor*) and slightly more resistance against bacteria (*Bacillus sp.*), within the experimental conditions adopted.

Introduction

In our earlier research article (Febrianto et al. 1999), we studied the effect of modification of *trans*-1,4-isoprene rubber (TIR) with maleic anhydride (MAH)

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that is MTIR, either as matrix or as compatibilizer on the physical, mechanical, and flow behaviour of TIR-wood flour (WF) composites. It was found that the presence of WF in the TIR-WF composite worsened the tensile and several other physical properties (water absorption and thickness swelling). However, when MTIR was added to the composite as compatibilizer, improvements in tensile properties and dimensional stability was noted. The scanning electron microscopy (SEM) observation demonstrated that interaction and adhesion between TIR and WF could be improved by the use of MTIR as compatibilizer. The improved physical and mechanical properties presumably resulted from the esterification between anhydride groups of MTIR and OH-groups of WF.

Furthermore, the mechanical properties of composites are considered to be affected by processing method and kneading conditions (such as mixing temperature, mixing time and rate of rotation) (Sean et al. 1991 and Takase and Shiraishi 1989). In the earlier publication (Febrianto et al. 1999), the MTIR was prepared in advance, and it was added to WF/TIR mixture during blending. This process may be referred to as *two-step* process. However, if the process is completed in *one-step* or *continuously*, it may be termed a shortened or simplified process.

Hence, in the present study, the possibility of the occurrence of grafting between MTIR and WF through esterification was investigated. In addition, the mechanical and flow properties of the TIR-MTIR-WF composites under various kneading conditions was studied. Further studies were also conducted to compare the mechanical properties as well as the biodegradability of WF-TIR composites prepared from both the *two-step* and the *one-step* process at several MAH-concentration.

Experimental

Materials

TIR was supplied by the Japan Synthetic Rubber Co., Ltd., while MAH was purchased from Nacalai Tesque Inc. and used as received. The filler used was WF that passed a 200 mesh sieve and corn starch (CS).

Preparation of MTIR

TIR and 0 ~ 40% MAH were reactively blended in the melted state in the kneader (Toyo Seiki Labo-Plastomill LPM 18-125) at 150 °C, the rotational speed was 30 rpm while reagents were introduced (3 min). Then the mixing was continued for 7 min at 70 rpm. The resulted MTIR was then used as a compatibilizer of the TIR-WF and TIR-CS composites.

Compounding the composites

The methods of processing composites were as follows:

1. *Two-step* process: MTIR was prepared in advance and added to the filler and TIR. This mixture was placed into a kneader which had been preheated to 80 ~ 150 °C and was rotated at 30 rpm for 5 min. After charging, the rate of rotation was increased to 30 ~ 110 rpm and kneading was continued for 3 ~ 25 min. The amount of matrix and WF were 24 grams, being enough to fill up the mixing chamber and causing torque while kneading, and the ratio of TIR-MTIR-WF was 4:1:5.
2. *One-step* process: The prescribed amounts of TIR, WF, and MAH were hand-mixed. This mixture was placed into a kneader, which had been preheated at

80 °C and rotated at 30 rpm for 5 min. After charging the rotation rate was increased at 90 rpm and kneading was continued for 5 min. The total kneading time was 10 min. The concentration of MAH applied was 0 ~ 20%, and the ratio of TIR and WF was 1:1. A control composite, containing only TIR and WF was also prepared.

Preparation of composite sheet

Kneaded samples were molded into sheets by hot pressing with a Toyoseiki 10 T bench. The prescribed amounts of kneaded samples (5 ~ 7 grams) were placed between a pair of thick teflon sheet with a 0.3 mm thick spacer. The temperature of the hot pressing was 120 °C, and subjected to 0 ~ 50 and 150 kgf/cm² pressure for 2 and 0.5 min., respectively. After subsequent cold pressing at the same pressure for 30 s, the sheets were then cooled at room temperature.

Tensile test

Strips samples 80 × 5 × 0.3 mm were prepared from composite sheets. Tensile tests were made on these strip samples with a Shimadzu Autograph DCS-R-500. The measurements were made with span lengths of 40 mm and cross head speed 10 mm/min in a room adjusted to 20 °C and 60% R.H. The average value of tensile strength, breaking elongation and Young's modulus were obtained automatically from ten repeated measurements. The average value of tensile strength, breaking elongation, and Young's modulus were obtained from ten repeated measurements.

Viscosity measurement

A flow tester (Shimadzu CFT-500 C) was used for the melt viscosity measurement. Approximately 1 ~ 1.2 grams of the kneaded samples after being dried at room temperature in *vacuo* for 48 hours were prepared for measurement, being made at 200 °C under a constant load of 100 kgf.

Flow test

Thermal flow temperature of the blended samples was measured by the flow tester. The measurement was made at constant heating rate of 10 °C/min and under compressive load of 100 kgf.

Water absorption test

The test procedure used for the water absorption test is JIS A 5908 with some modifications. Square samples of 50 × 50 × 0.3 mm size were prepared from the composite sheets. The water absorption test was made on these square samples in three replications. The samples were dried overnight in the oven at the temperature of 50 °C, then stored in a desiccator. The weight of the samples was measured in a room adjusted to 20 °C and 60% R.H. Subsequently, the samples were dipped in water for 24 hours and conditioned in a room adjusted to 20 °C, 60% R.H. for 48 hours. Then, the samples were wiped and weighed. Finally, the samples were dried in a 50 °C oven to constant weight. Water absorption was calculated.

Soxhlet extraction of kneaded samples and subsequent measurements of weight loss

The obtained kneaded samples were dried and accurately weighed. After Soxhlet extraction with hot xylene for 48 hours, the extracted samples were dried and

weighed again to obtain the weight decrease percentage of the samples. The weight loss is due to unreacted TIR soluble in xylene.

Infrared spectroscopic measurement

A Shimadzu FTIR 8600PC apparatus was used for infrared spectroscopic measurements. The KBr disk technique was employed for the analysis. The amount of sample and KBr were 0.002 g and 0.2 g, respectively. FTIR spectra were obtained in the $400 \sim 4600 \text{ cm}^{-1}$ range with a 4 cm^{-1} resolution.

Biodegradability test

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1. Decay test: Mono-culture decay tests were conducted according to Japan Preserving Association (JWPA) Standard No.3-1992, using a brown-rot fungus *Tyromyces palustris* (Berk.et.Curt.) MUrr.-FFPRI-0507, and white-rot *Coriolus versicolor* (L.ex FR.) Quel.-FFPRI-1030. The samples ($10 \times 10 \times 0.3 \text{ mm}$) were sterilized in gaseous ethylene after measuring their original dry weight. The samples in the glass jars were kept at $28 \text{ }^\circ\text{C}$ and exposed to fungal attack for 12 weeks. The samples were then cleaned and dried in a $50 \text{ }^\circ\text{C}$ oven to constant weight. The extent of attack was determined based on the percent of weight loss.
2. Termite test: The samples ($10 \times 10 \times 0.3 \text{ mm}$) were subjected to subterranean termites in accordance with JWPA Standard No. 12-1992. *Coptotermes formosanus* Shiraki was used for the test. Forty-five workers and five soldiers of termites were subjected to the forced-feeding test with 20 mesh sand as medium. The samples in glass bottle were kept in the dark room at $28 \text{ }^\circ\text{C}$, and 80% R.H. for 3 weeks after measuring their original dry weights. Then the samples were cleaned and dried in a $50 \text{ }^\circ\text{C}$ oven to constant weight. The weight loss of each sample was calculated at the end of the test.
3. Composting test: Square samples of $20 \times 20 \times 0.3 \text{ mm}$ size were prepared from the composite sheets. The samples were dried overnight in the oven at a temperature of $50 \text{ }^\circ\text{C}$, then stored in desiccator. The weight of the samples was measured. After wrapping with 40 mesh stainless-wire, the samples were placed into the composting chamber (Nature Pocket NS-01, Shizen Koubou Inc.) for 8 weeks. The chamber capacity was 20 litres containing 10 litres sawdust and 200 ml bacteria suspension (*Bacillus sp.*). The chamber temperature was $40 \text{ }^\circ\text{C}$. The food waste supplied was about 1 kg every 2 days. In order to supply oxygen to the aerobic bacteria, the chamber was automatically stirred for 10 min every 4 hours. Then, the samples were cleaned and dried in a $50 \text{ }^\circ\text{C}$ oven to constant weight. The weight loss of the samples was calculated at the end of the test.

Results and discussion

Evidence for the occurrence of grafting between WF and MTIR

In order to impart more outstanding physical and mechanical properties to the MTIR-WF composites compared with those found in the TIR-WF composites, the possibility of the occurrence of grafting between WF and MTIR through esterification is considered. This grafting may improve the adhesion between the WF and the MTIR. In order to confirm the finding that chemical bonding takes place in the MTIR-WF composites, several composites were prepared for both MTIR and TIR composites by changing the weight ratio of WF over MTIR or TIR matrices. The MTIR used was MTIR•20 (MTIR was prepared from TIR and 20% MAH).

First, WF and TIR or MTIR were mixed in various proportions and then blended by a kneader. Each of the composites was extracted with hot xylene for 48 hours and the extracted fraction was measured as weight decrease percentages. The results are shown in Table 1 together with the theoretical values obtained on the assumption that no grafting occurs during kneading.

As the Table shows, the weight decrease value of TIR-WF composites agree well with the theoretical values, whereas those of MTIR-WF composites fall short of the theoretical values. That means that TIR can be completely removed from the TIR-WF composites by extraction with xylene, whereas part of the MTIR remains within the unextracted residue when the MTIR-WF composite is subjected to extraction. These results could be further confirmed by the FTIR spectroscopic analysis of the unextractable residue as shown in Fig. 1.

The residue from the TIR-WF composite reveals a spectrum very similar to that of WF and does not reveal peaks attributable to TIR. This means that TIR was completely removed by extraction. The peak at around 1790 cm^{-1} corresponds to C=O of WF. In the spectrum for the residue of MTIR-WF, this peak was bigger

Table 1. Weight decrease percentages of each of the composites after extraction with hot xylene for 48 hours

TIR: WF	Weight decrease (%)		
	MTIR-WF composite	TIR-WF composite	Theoretical value ¹⁾
8:2	76.7	80.3	80
7:3	67.6	70.9	70
6:4	57.9	60.5	60
5:5	48.2	51.3	50

¹⁾ Assumes TIR and MTIR both are completely soluble in xylene

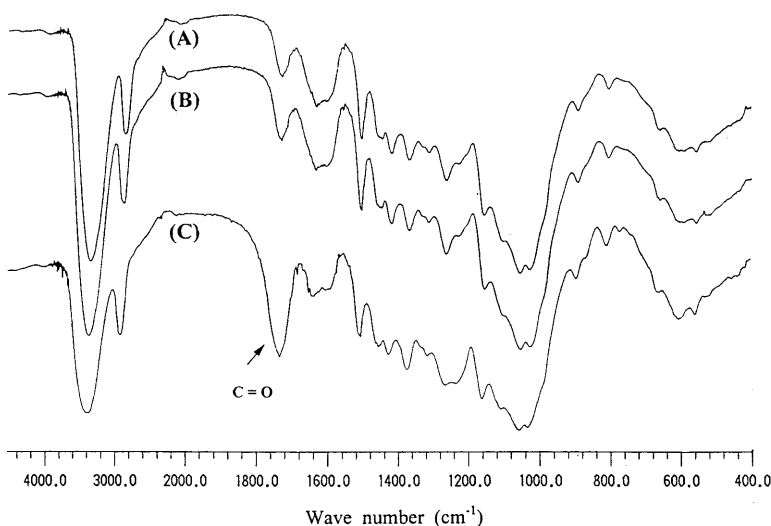


Fig. 1. Infra red spectra of related samples (A: WF; B: xylene unextractable residue from 50:50 TIR-WF blend; C: xylene unextractable residue from 50:50 MTIR-WF blend)

than in the case of WF or the residue of TIR-WF spectra indicates perhaps that this peak overlaps with C=O of MAH. Therefore, from these facts, it can be concluded that grafting through esterification has been attained between the hydroxyl groups of WF and the acid anhydride group of MTIR, during kneading under heating. This grafting makes the wood surface hydrophobic resulting in a superior adhesion between WF and the matrix polymer phase.

Effect of processing conditions on the tensile and flow properties of TIR-MTIR-WF composites

Effect of rotation rate

In this study, keeping the mixing temperature at 80 °C and mixing time at 10 min, the rate of rotation of the kneader was varied from 30 ~ 110 rpm. The results of the tensile properties are presented in Fig. 2.

It is quite apparent from Fig. 2. that increasing the rate of rotation results in an increase in the tensile strength and the breaking elongation of the composites obtained. It has been demonstrated that the length of the fibers (refiner ground pulp) decreases and the structure of the fibers diminishes as the fibers are finely pulverized at a rotation rate increasing from 30 to 90 rpm (Takase and Shiraishi 1989). A high rotation rate leads to better dispersion (homogeneity) of the fibers within the composites, improving adhesion between the matrix polymer and filler. In our case a similar effect occurred in the composites of TIR-MTIR-WF. A maximum tensile strength value was found at 90 rpm. The breaking elongation value shows a steady increase with the rate of rotation. At 90 rpm, the values of tensile strength, breaking elongation, and Young's modulus are 22.60 MPa, 12.6%, and 507 MPa, respectively.

Concerning the melt viscosity of the composites obtained, an increase in the rotation rate significantly reduces the melt viscosity of the composites (Fig. 3). On the other hand, the value of the flow temperature remained almost constant with an increase in the rotation rate up to 90 rpm, and further decrease beyond this point. Generally, the melting temperature of the polymers is lower when their molecular weight declines and their apparent viscosity decreases. As temperature

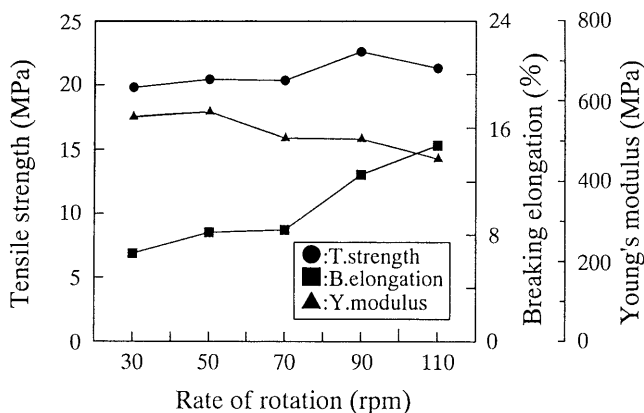


Fig. 2. Effect of rotation rate on tensile strength, breaking elongation and Young's modulus of the composites (MTIR is prepared from TIR and 10% MAH; WF = 50%; TIR/MTIR•10 = 4/1)

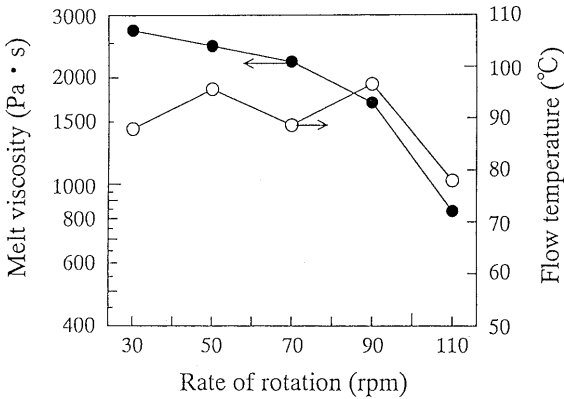


Fig. 3. Effect of rotation rate on melt viscosity and flow temperature of the composites (MTIR is prepared from TIR and 10% MAH; WF = 50%; TIR/MTIR•10 = 4/1)

increases, the links between the particles are ruptured. This in fact, produces a decrease of the viscosity of the system.

Effect of mixing time

The effect of mixing time on the tensile and flow properties was evaluated in the range of 8 ~ 30 min. The mixing temperature and the rate of rotation were fixed at 80 °C and 90 rpm. The results obtained on the mechanical properties are shown in Fig. 4.

Figure 4 reveals that initially the tensile strength increases with mixing time, but with increased mixing time (more than 10 min) it remains constant or decreases slightly. The maximum tensile strength is achieved after 10 min of mixing. The quality of dispersion controls the performance of short-fiber composites. Increasing mixing time from 8 to 10 min provides good dispersion to the mixture. Breaking elongation and Young's modulus both increase as the mixing time increases from 8 to 20 min. They decline at >20 min of mixing time. These observations can be attributed to homogeneous WF distribution and to fine pulverization after 10 min of mixing.

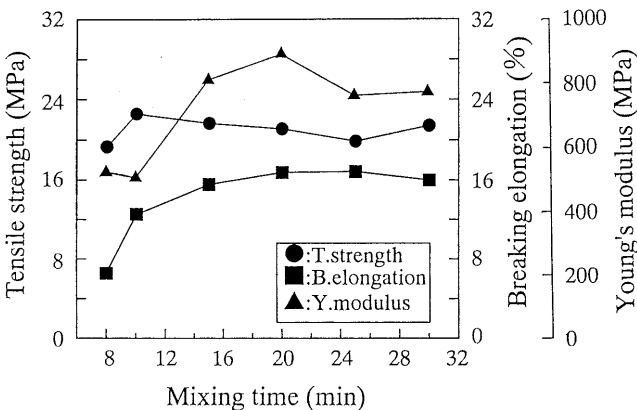


Fig. 4. Effect of mixing time on tensile strength, breaking elongation and Young's modulus of the composites (MTIR is prepared from TIR and 10% MAH; WF = 50%; TIR/MTIR•10 = 4/1)

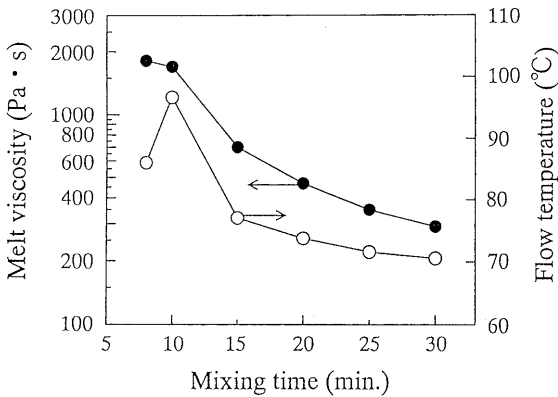


Fig. 5. Effect of mixing time on melt viscosity and flow temperature of the composites (MTIR is prepared from TIR and 10% MAH; WF = 50%; TIR/MTIR•10 = 4/1)

The melt viscosity and flow temperature of the composites obtained from various mixing time are presented in Fig. 5. It is observed that the influence of the mixing time on the melt viscosity and flow temperature of the composites is significant. Prolonging the mixing time past 10 min results in continuous decrease of the melt viscosity and flow temperature. The decrease of this values can be attributed to the matrix degradation.

Effect of mixing temperature

The effect of mixing temperature on the tensile and flow properties was studied by varying the mixing temperature between 80 ~ 150 °C, while maintaining the mixing time and the rotation rate at 10 min and 90 rpm, respectively. The results of the tensile properties are illustrated in Fig. 6.

As for tensile strength, it slightly increases with increasing mixing temperature, yielding a weak maximum at 120 °C. This phenomenon occurs with the Young's modulus. The breaking elongation increases linearly with mixing temperature, yielding its maximum at 140 °C. Tensile strength, breaking elongation, and Young's modulus all are in the range of 20.6 ~ 23.4 MPa, 10.4 ~ 15.7% and 507 ~ 917 MPa, respectively. In fact, in short-fiber composites, the fibers aspect ratio, the quality of dispersion, and the interface between fiber and polymer directly control the performance of the composites. In this case a high degree of

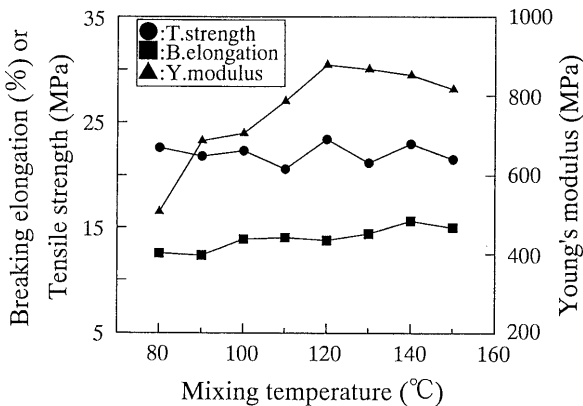


Fig. 6. Effect of mixing temperature on tensile strength, breaking elongation and Young's modulus of the composites (MTIR is prepared from TIR and 10% MAH; WF = 50%; TIR/MTIR•10 = 4/1)

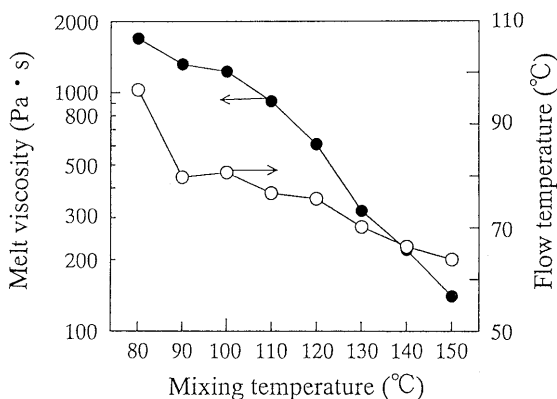


Fig. 7. Effect of mixing temperature on melt viscosity and flow temperature of the composites (MTIR is prepared from TIR and 10% MAH; WF = 50%; TIR/MTIR•10 = 4/1)

homogeneity and quality of dispersion of the WF in the MTIR-TIR matrix have been achieved at 120 °C.

The melt viscosity and flow temperature results are given in Fig. 7. It is obvious that the melt viscosity and flow temperature are affected to a great extent by the mixing temperature. The higher the mixing temperature, the lower the melt viscosity and the flow temperature. This phenomenon can be attributed to a decrease in the molecular weight due to the degradation of the matrix. Indeed, it was observed that the color of the mixture became darker when the mixing temperature increased.

Effect of MTIR addition on the tensile properties of TIR-WF, and TIR-CS composites

In the previous part the best conditions for the preparation of TIR-WF composites have been determined, that is 120 °C, 30~90 rpm for 10 min. In the present study a new filler corn starch (CS), was tested and MTIR was evaluated as a compatibilizer of the TIR-CS composites. MTIR was prepared from TIR and 10% MAH. The TIR-MTIR-WF composites were also prepared for the purpose of comparing the performance of CS as a filler. The results are shown in Fig. 8.

From Fig. 8 it is clear that the tensile strength of the TIR-MTIR-WF composite is higher than the tensile strength of consisting of TIR-MTIR-CS composites. The difference in strength values obtained can presumably be attributed to the difference in properties between WF and CS. Wood flour is a composite material mostly composed of cellulose (40–50%), hemicellulose (20–30%), and lignin (20–30%) with small quantities of inorganic compound and extractives. Cellulose consists simply of D-glucose units linked by the 1,4'- β -glycoside bond. Several thousand glucose units are linked to form one large molecule, and different molecules can then interact to form a large aggregate structure held together by hydrogen bonds. On the other hand, starch is a polymer of glucose in which monosaccharide units are linked by the 1,4'- α -glycoside bonds. Starch can be separated into two fractions: a fraction soluble in cold water, called *amylopectin*, and a fraction insoluble in cold water, called *amylose*. Amylose, which accounts for about 20% by weight of starch, consists of several hundred glucose molecules linked together by 1,4'- α -glycoside bonds (Mc Murry, 1992). Hence, filler with a higher degree of polymerization can result in greatly strengthened composites. The tensile strength and breaking elongation of both composites, TIR-MTIR-WF and TIR-MTIR-CS, increase when the MTIR content

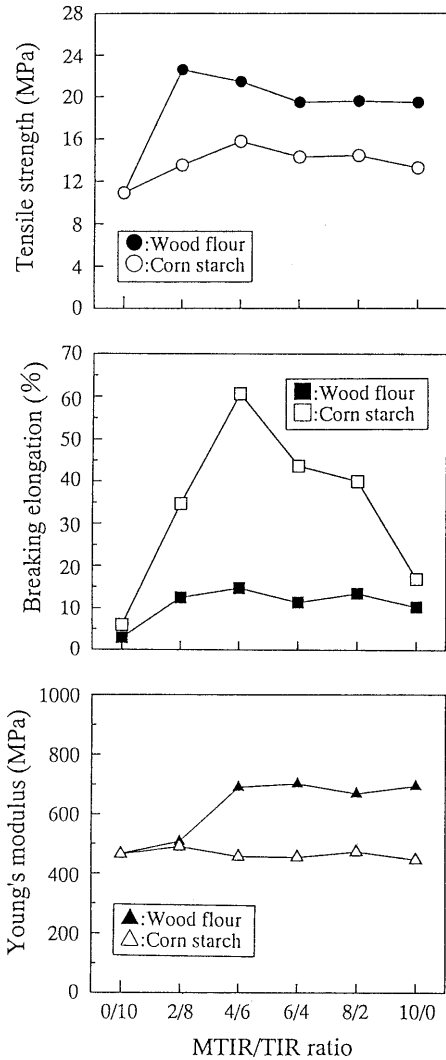


Fig. 8. Relationship between MTIR•10/TIR ratio and tensile properties of the TIR-MTIR-WF and TIR-MTIR-CS composites

increases. An addition of 10% MTIR to the whole TIR-MTIR-WF composite results in the highest tensile strength. As for the TIR-MTIR-CS composite, the highest tensile strength value is obtained at 20% MTIR level. The values of tensile strength are 22.6 MPa and 14.8 MPa, respectively, for TIR-MTIR-WF and TIR-MTIR-CS composites at this point. These tensile strength values are 2.1 and 1.6 times higher than the control ones. So, MTIR acts as a compatibilizer in both types of composites. The Young's modulus of the TIR-MTIR-WF composites also improves by addition of MTIR compatibilizer, while for the TIR-MTIR-CS the composite is unchanged. Further addition of the MTIR results in almost no additional improvement.

As to the action of the compatibilizer, MTIR would presumably be localized at the interface between WF or CS and TIR matrix, improving the adhesion between these two composites. Chemical bonding between MTIR and WF or CS would also play an important role in improving mechanical properties of the composites.

The physical and mechanical properties of the composites prepared using several processing methods at different MAH concentration

So far, all composites have been prepared by the *two-step* process. It is quite possible that the properties of composites, particularly tensile properties, may vary with difference in the processing method. Therefore, in this part, the effect of the processing method on the mechanical and physical properties of the composites was investigated at different MAH concentrations. In the *two-step* process, the ratio of TIR to MTIR was 4:1, and MTIR was prepared from various MAH concentrations ranging from 0 ~ 40%. The ratio of matrix to filler was 1:1. In *one-step* process, WF-TIR and MAH were hand-mixed before charging to the kneader. The concentration of MAH used was 0 ~ 20%. The control composite containing only TIR and WF was also prepared. Kneading temperature, kneading time, and rotation rate were 80 °C, 10 min, and 90 rpm, respectively.

In the case of the TIR-MTIR-WF composites prepared using *two-step* process, the MAH-concentration was found to greatly affect tensile properties (Fig. 9). Increasing the MAH-concentration up to 10% increases tensile strength and breaking elongation; at higher MAH-concentrations both properties, gradually decrease. Furthermore, the Young's modulus value also improves with addition of MAH concentration and shows a maximum value at a MAH concentration less than 5%. Based on these observations, the maximum of tensile strength and breaking elongation have been achieved at MTIR•10 and MTIR•1 (MTIR prepared from TIR and 10% MAH, and TIR and 1% MAH).

As for the TIR-MAH-WF composites prepared using *one-step* process, the tensile properties of the composites are also greatly affected by the MAH-concentration (Fig. 10). Tensile strength and breaking elongation increase with rising MAH- concentration and remain constant or decrease at >8% MAH addition. The Young's modulus also increases with addition of MAH, yielding the maximum at 4% MAH addition. The typical comparative stress versus strain curve of TIR-WF and TIR-MAH-WF are given in Fig. 11. The stress-strain curve of TIR-WF belongs to soft and weak materials type. It was indicated by low tensile properties (tensile strength, breaking elongation, and Young's modulus). However, addition of MAH to the composite helped to transfer stress from the matrix to the fiber, and shifted the mechanical characteristics of the system towards a stiffer and

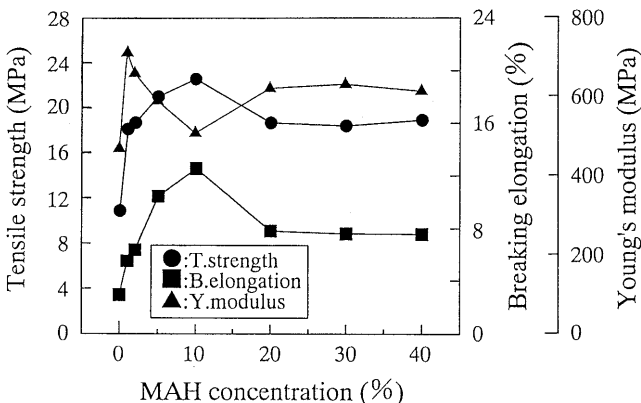


Fig. 9. Effect of MAH concentration on the tensile properties of the TIR-MTIR-WF composites prepared using the *two-step* process (TIR/MTIR/WF = 4/1/5)

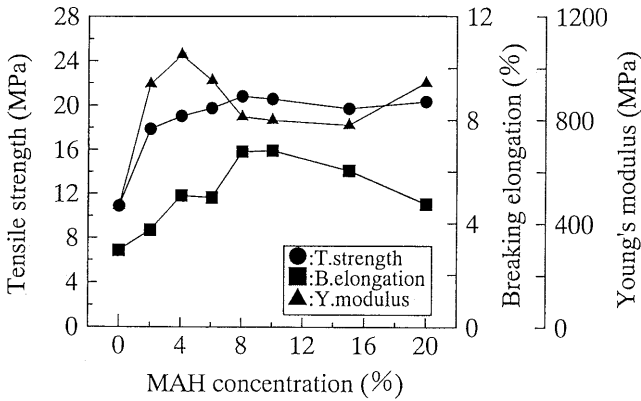


Fig. 10. Effect of MAH concentration on the tensile properties of the TIR-MAH-WF composites prepared using the *one-step* process (TIR/WF = 5/5)

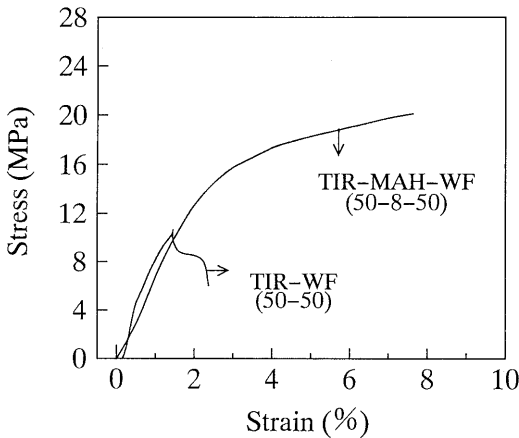


Fig. 11. Characteristic stress-strain curve for TIR-WF and TIR-MAH WF composites

stronger material with higher breaking elongation. These results suggest that during kneading maleoylation between MAH and TIR, and esterification between acid anhydride groups of MAH and the OH-groups of WF occurred even at low temperature (80 °C). It has been reported that esterification occurs easily between maleic anhydride and wood when mixed and heated at more than 60 °C even without catalyst and solvent (Matsuda et al. 1984). The composite prepared using the *one-step* process possesses lower tensile strength and breaking elongation values compared to the composites prepared using the *two-step* process.

Figure 12 reveals the effect of MAH concentration on the water absorption of TIR-WF composites prepared using the *one-step* process. It is obvious that addition of MAH to the composite drastically reduces water absorption up to 8% addition and remains constant beyond this point. This phenomenon might be attributed to some of the hydrophilic OH-groups of WF reacting with acid anhydride of MAH to form ester linkages when kneaded at 80 °C for 10 min, followed by hot-pressed at 120 °C for 2.5 min.

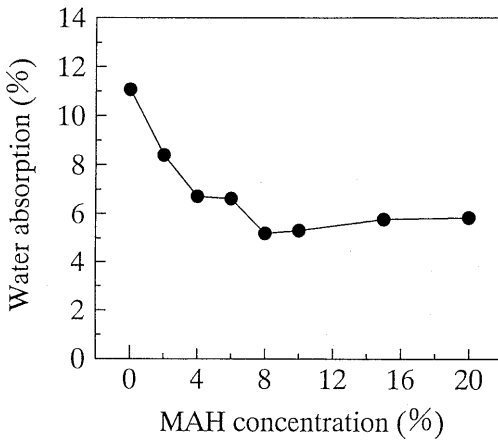


Fig. 12. Effect of MAH concentration on the water absorption of the TIR-MAH-WF composites prepared using the *one-step* process (TIR/WF = 5/5)

Effect of MTIR compatibilizer on the biodegradability of TIR-WF composites

Weight loss of the TIR-WF and TIR-MTIR-WF composites after 3 weeks, 8 weeks, and 3 months exposed to subterranean termite *C. formosanus*, bacteria *Bacillus sp.*, and white and brown-rot fungi *C. versicolor* and *T. palustris*, respectively, are presented in Table 2.

The data presented in Table 2 showed that addition of MTIR compatibilizer slightly affected the resistance of the composites against brown-rot *T. palustris* and white-rot *C. versicolor*, subterranean termite *C. formosanus*, and bacteria *Bacillus sp.* The TIR-WF composites with MTIR compatibilizer had slightly less resistance against brown-rot *T. palustris* and white-rot *C. versicolor*, subterranean termite *C. formosanus* than TIR-WF composites. On the other hand, the addition of MTIR compatibilizer slightly increased the resistance of the composite compared to the TIR-WF against *Bacillus sp.* The TIR-WF with MTIR showed similar resistance as the TIR against *Bacillus sp.*, but the TIR-WF without MTIR had less resistance than the TIR against *Bacillus sp.* Therefore, it can be concluded that esterification between OH-groups of WF and acid anhydride groups of MAH increased the resistance of composites againsts *Bacillus sp* within the experimental conditions adopted.

Table 2. Weight loss of TIR-WF and TIR-MTIR-WF composites after exposure to subterranean termite (*C. formosanus*), white-rot fungus (*C. versicolor*), brown-rot fungus (*T. palustris*), and bacteria (*Bacillus sp.*)

Samples	Weight loss (%)			
	<i>C.formosanus</i>	<i>C.versicolor</i>	<i>T.palustris</i>	<i>Bacillus sp.</i>
TIR	0	0.54	0.82	0
TIR-WF	14.34	1.10	1.22	1.21
TIR-MTIR•10-WF	15.19	1.47	1.32	0

Note: TIR-WF = 50-50; TIR-MTIR•10-WF = 40-10-50

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